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A simple two-crystal spectrometer made for investigating crystal lattices

By Gösta Brogren

With 4 figures in the text

Crystal lattices, used within X-ray spectroscopy, are obtained by cutting out a plane-parallel plate from a crystal. The surface must be parallel to a suitable atomic plane. When this plane is also a cleavage surface, it is possible to obtain a plate with good surfaces by cleaving it out from a crystal. In other cases the surface must be ground and polished. During this procedure it is smoothed out, because all the irregularities are filled by a mixture of crystal powder, and polishing materials. The surface is made optically plane, but when the crystal plate is used as an X-ray lattice, the surface layer causes a strong line-broadening and decreases the resolving power.

We have constructed a spectrometer in order to study how a removal of this surface layer will influence the resolving power of different crystal lattices of quartz and calcite. As the principles of the two-crystal spectrometer are well described previously[1], we will only give a brief description of its construction.

The Construction

The construction appears from Figs. 1, 2, and 3. The spectrometer is built on a circular steel plate *I* having a diameter of 55 cm and a thickness of 2.5 cm, its two surfaces being ground plane and parallel. In the centre the cone-system *D* from a "tube spectrometer" is mounted with its axis perpendicular to the surface of the steel plate. The outer cone is screwed to the bottom of the circular plate. The male cone is pressed into the outer one by counterbalance *K*, which presses it up by means of the lever system in Fig. 2. This male cone, carrying the analysing crystal *B*, is provided with a very accurate circular scale S_B, which is read on two measuring microscopes mounted diametrically opposite. They are provided with ocular micrometers, which make it possible to determine the angular position with an accuracy of $0.2''$. The fine adjustment of the scale S_B is made by means of the micrometer screw *E*, which has a screw pitch of 0.5 mm. and whose drum is graduated in 500 divisions. A displacement of one division (0.001 mm) on the micrometer screw causes a rotation of the male cone of $0.800''$. The X-ray tube *C* is of the usual construction. The electrons from the filament are focussed on to the target by means of a

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[1] A. H. Compton and S. K. Allison. "X-rays in Theory and Experiments".

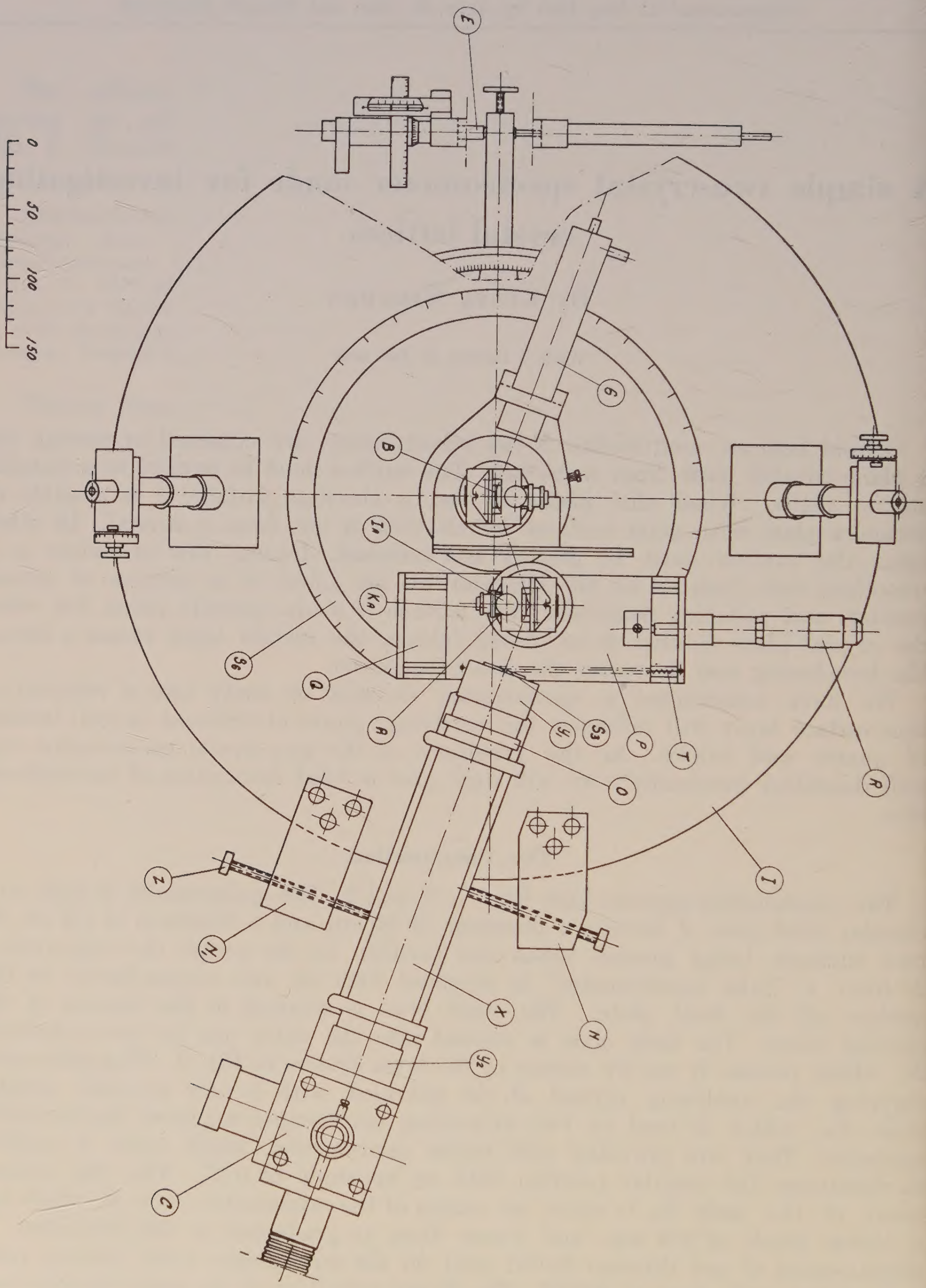

Fig. 1. The spectrometer from above.

focussing cylinder. The target has an inclination of only 10° in order to give the focal spot as small vertical extension as possible. The radiation passes out through a circular hole, 8 mm in diameter covered with an aluminium foil, 0.04 mm thick. The X-ray tube is mounted on a revolvable arm M movable around axis H. This arm consists of a plate of hardened steel, 60 mm broad, 10 mm thick, and 260 mm long, which is ground plane-parallel. It carries also a brass tube X, which is 300 mm long and has a wall thickness of 3 mm. On the outside it is turned so that the diameter is 50 mm. The end surfaces hold the two horizontal slits S_1 and S_2, the widths of which can be varied between 0 and 6 mm. On the ends there are two external identical end pieces Y_1 and Y_2, which hold the two vertical slits S_3 and S_4. Their breadths can be varied from 0 to 15 mm. The brass tube lies in two U-shaped holders retained there by two strait springs. On its outside there is a cylindric ring O furnished with a knob. The knob lies between the two screws L, which makes it possible to turn the tube around its own axis. The position of the X-ray tube can be read on a circular scale at H (not to be seen in the figure). In order to make fine adjustments possible, there are two clamps N furnished with graduated screws. The arm carrying the X-ray tube lies between the ends of these.

The monochromator crystal A is mounted on a crystal table of the same type as those used in plane crystal spectrometers. It is placed on a slide running on ball-bearings, consisting of the two plates P and Q. The upper plate is movable on three balls running in two parallel grooves in the lower one. The forms of these are obvious from Fig. 2. The plates are made of hardened steel, and carefully ground and polished.

The upper plate P can be displaced by the micrometer screw R (50 mm long) against which it is pressed by spring T. The slide is placed so that when adjusted in the zero position the midpoint of the crystal lies on the middle of the line between the axes of rotation of the X-ray tube and the B crystal. Its displacement occurs at right angles to this line. The maximum displacement (50 mm) allows an alteration of the glancing angle of 30°.

As the two crystal tables are of the type commonly used in plane crystal spectrographs, they are not drawn in detail. The very crystal holders (Fig. 3) are so made as to make it possible to exchange its crystal plates without destroying the adjustments. On the front side the holders have two angles U_1 and U_2. The lower U_1 is fitted with one ivory knob and the upper with two. Their tops are made semi-spherical. Against them two silver steel pins press which also have rounded tips. The crystal plate is placed between the knob. The pressure is caused by phosphor-bronze spring V mounted on the rear of the holder. In order to simplify the adjustments there is a rectangular hole cut out in the holder and the spring plate. The distance between the lower edge of the upper angle and the upper edge of the lower is 6 mm. The vertical distance between the ivory knobs is 10 mm, and the distance between the two upper ones is also 10 mm. As appears from Fig. 3 there is an opening in the upper angle that is 16 mm wide. The crystal plates to be investigated are 12 to 15 mm broad. The construction of the holder makes it possible to put in a crystal plate either from the side or from above.

The intensity of the X-radiation is registered by Geiger-Müller counter G. It has on its front side a horizontal slit (not drawn in the figure) the height of which can be varied from 0 to 6 mm. Its width is 15 mm. The position

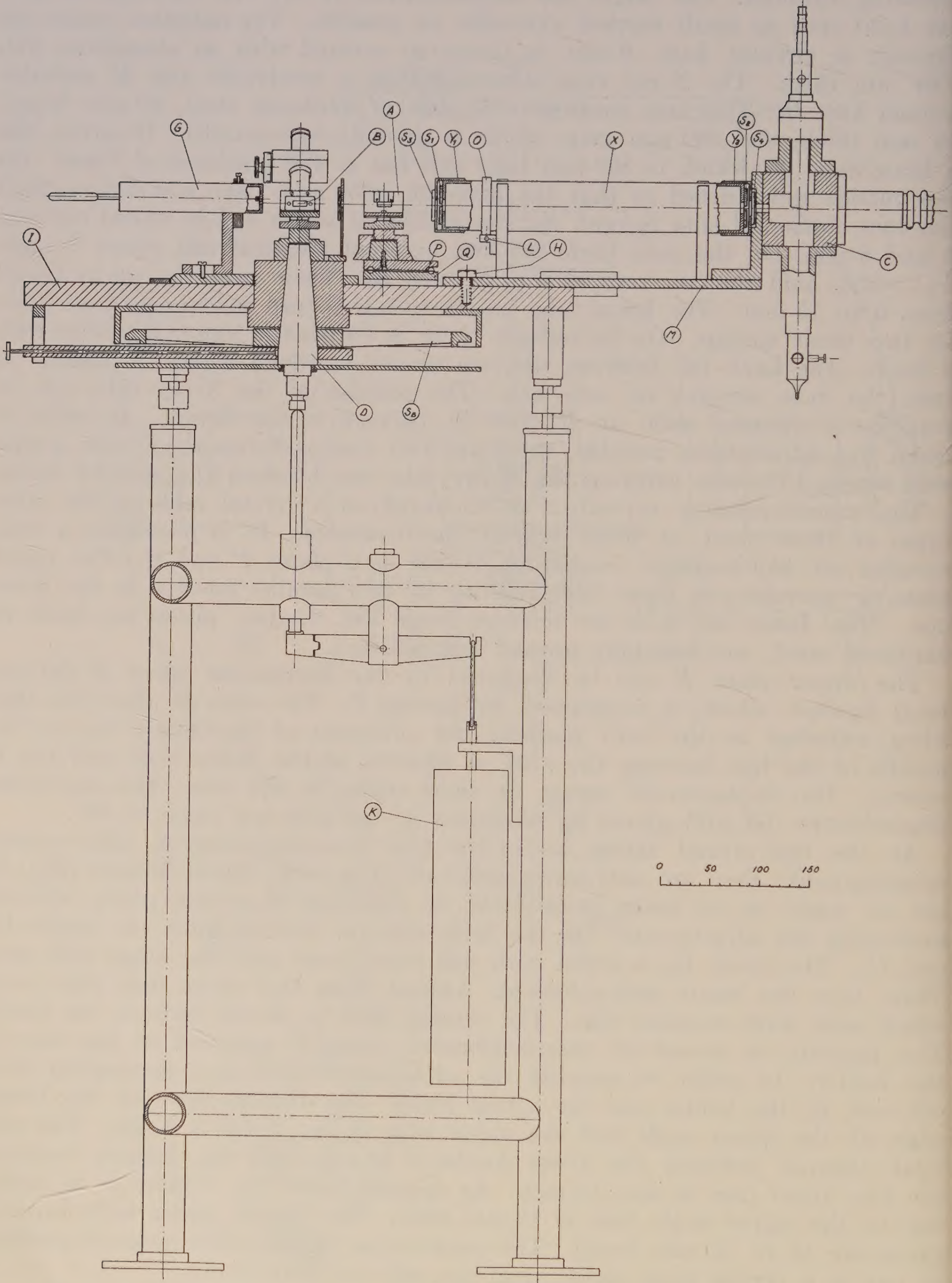

Fig. 2. Sideview of the spectrometer.

of the G-M counter is read on scale S_G. The set of apparatuses is built so that the focal spot, the axis of the brass tube X with the slit system, the midpoints of the free crystal surfaces and the midpoint of the slit of the G-M-counter are all in the same level.

In order to avoid secondary X-radiation in the room a lead shelter 3 mm thick is placed around the scale of the G-M-counter. It is covered by a brass roof also 3 mm thick. Between the two crystals there is an adjustable shutter preventing secondary X-rays from disturbing the registrations. The walls of the brass tube X are thick enough to absorb all radiation entering them.

Adjustments

The two-crystal spectrometer is adjusted as follows:

1. The surface of the steel plate I is adjusted horizontally by means of a water level.

2. The slide F is set at right angles to the line between the two axes of rotation B and H. Its position is adapted so as to cause the centre of the A crystal to lie on the normal through the centre of the line BH.

3. In the holder for the B-crystal there is placed a plane-parallel glass plate which is adapted parallel to the axis of rotation in the usual way. By using a centring microscope and a quartz loop, the surface that is pressed against the ivory knobs is adjusted so as to lie in the axis of rotation.

4. The X-ray tube and its arm are removed. A vertical slit is placed in the centre H of its axis of rotation. By reflecting the slit in the glass surface, its image is brought to coincide with the slit itself by turning the crystal cone. This gives us the zero position of crystal B. (As a slit we used a very thin vertical wire, which was illuminated.)

5. We insert a plane-parallel glass plate into the adjusted holder B, which is set to its zero position. Another plane-parallel glass plate of the same size is placed in the holder A, which is adjusted approximately parallel to B. The slide is set so that "crystal plane" A is above line BH. (As the crystal plane we denote the tangent plane of the three ivory knobs.) In this position the ends of the glass plates must be nearly in contact with each other. A telescope with a scale is placed on the normal to BH through the point where the glass plates nearly touch each other. In the telescope we can observe two images, one from each glass plate. By adjusting holder A (turning it around the cone and tilting it vertically by using the adjustment strew) the two images are brought to coincide. The crystal planes of the two holders are now parallel, i.e. that of holder A is parallel to the axis of rotation of holder B. As it was not possible to observe deviations from this coinciding less then 0.2 mm, and as the distance scale-glass-mirror was 4 000 mm, the deviation from parallelism is below $10''$.

6. In order to control that the slide displaces crystal A parallelly to itself, the image of the scale in the glass plate in A is observed with the telescope. The slide is moved 50 mm by means of the micrometer screw. The image is observed at the same time. During our control we could see no displacement of the image.

7. The two crystal holders are placed in the parallel position. A vertical slit is arranged in the axis of H as in 4. Holder A is moved by means of

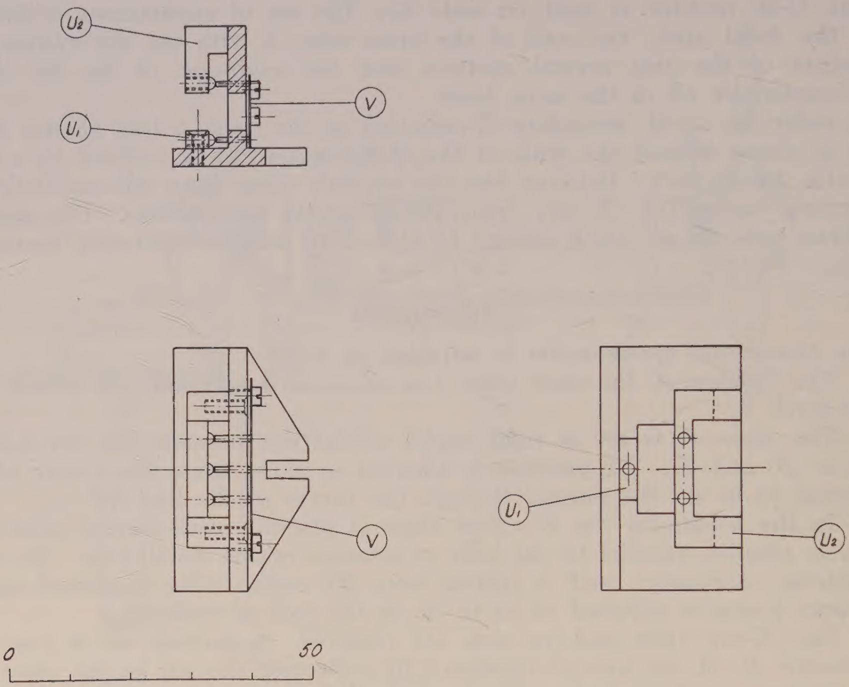

Fig. 3. The crystal holder.

the micrometer screw until the image in A coincides with the slit. This gives us the zero position of the slide on the screw scale. Then the glass plates are removed.

8. The slits S_1 and S_2, fixed to the ends of the brass tube X, are adjusted parallel and to the widths desired as follows: The outer end-pieces Y_1 and Y_2 carrying S_3 and S_4 are removed. Two combination gauge blocks of exactly the same height h mm are placed on a face-plate with tube X lying above them. The lower slit edges are removed, the upper ones are loosened so as to be displaceable, and at the end surfaces of the brass tube there are placed two other gauge blocks on the face-plate, both having the height $25 + h + 0.5\,d$ mm. The upper slit edges are brought into contact with these along their whole lengths and screwed on. A gauge block having the height d is placed in slit S_2 and the other slit edge is put on. Both edges are forced to be in contact with the gauge block along their whole lengths, and the second edge is fixed. Tube X is placed in its holders in front of the X-ray tube. By means of gauge blocks or an adjustment sliding caliper the slit edge of S_1 is made parallel to the surface of steel plate I. The tube is fixed in this position with the two screws L. S_1 is given the desired height d mm in the same way as described concerning S_2.

The vertical slits S_3 and S_4 are adjusted in a similar way. The outer end-piece Y_1 facing the crystals is put on; one of the edges of S_3 is fixed to it and adjusted perpendicular to the plane steel plate (by means of gauge blocks and an accurate steel square). In this position the end piece is screwed firmly to the brass tube. One of the two screws *L* is loosened so that the brass tube can be taken out of its holders. The second end piece Y_2 is put on. By starting from the above slit edge of S_3, the two slits S_3 and S_4 are parallelled to this and given the desired widths by means of the face-plate and gauge blocks in the same way as when adjusting S_1 and S_2. Tube *X* is replaced in its holders and fixed there in the correct position by pulling in screw *L* previously loosened. This procedure makes the two slit systems perpendicular to each other.

The slit of the G-M-counter is adjusted parallel to the basic steel plate and given the desired width by means of gauge blocks and an adjustment sliding caliper as above.

The adjustment of the slit system S_1 to S_4 can be controlled by fixing a plane parallel glass plate between the edges of S_3. The X-ray tube is adjusted on the glancing angle 0°. Another plane parallel glass plate is placed in crystal holder *A*. By means of a scale and a telescope it is controlled that the images of the scale in the the two glass plates lie on the same height.

X-ray adjustments

9. By means of pinhole-camera recordings, the correct height of the focal spot is controlled.

10. As an *A*-crystal a calcite crystal with a cleavage surface is placed into the holder. Its surface must have good qualities and be very smooth. Slide *F* is set so as to obtain the correct glancing angle. The X-ray tube is turned to the right position. A knife-edge shutter is placed in front of the *A*-crystal so that only a small region around the centre of the free surface can reflect the radiation. The reflected beam passes through the empty *B*-crystal holder and falls into the G-M-counter. The X-ray tube is turned by the two adjustment screws in the two clamps *N* until a maximum intensity is registered by the counter.

As a *B*-crystal another calcite crystal of the same good quality as that of *A* is inserted. The spectrometer is set to the (1.1)-position, registering the $K\alpha_1$-line of the target material (in our case copper). A control of the position of the X-ray tube is made so as to attain the maximum intensity of the $K\alpha_1$-line.

11. The calcite crystal in the *B*-position is removed, and the first of the two crystals to be investigated is placed in the holder. The position of the top of the $K\alpha_1$-line is determined. The crystal is taken out from the holder, turned 180°, and replaced again. These two positions in the holder are designated *a* and *b* in Fig. 4. The position of the top of the $K\alpha_1$-line is determined again. As the investigations comprise crystal plates where the surfaces are ground and polished parallel to a suitable atomic plane, there is a great risk that the surface and the atomic plane in question will not be parallel. Fig. 4e shows how the atomic planes are situated in the holder in positions 4a and 4b when this is the case. When the position of the crystal plate is changed from 4a to 4b, the crystal cone must be turned twice the angle between the surface and the atomic plane if maximum intensity is to be registered

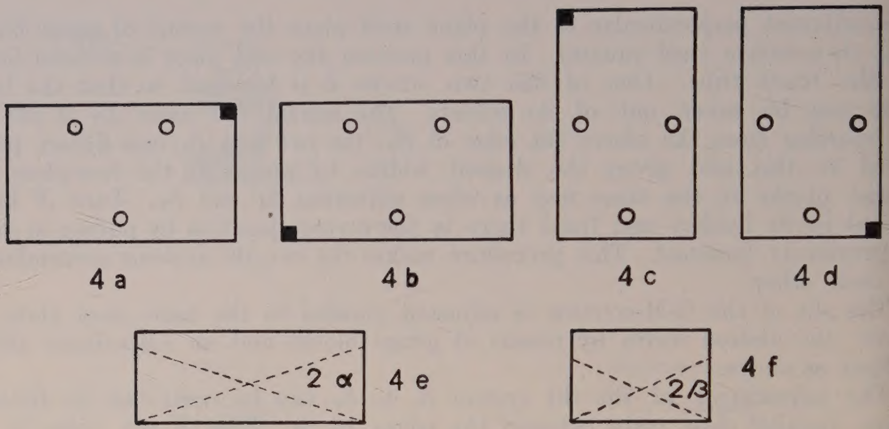

Fig. 4. The adjustment of the crystal plate. *a*, *b*, *c* and *d* are the four different positions of the crystal plate. The small circles are the ivory knobs. *e* is a cross section of the crystal plate in positions *a* and *b*. The locations of the atomic plane in these two cases are shown by means of the dotted lines. *f* is the corresponding cross section of positions *c* and *d*.

again. Hence, the difference between the 4a and 4b positions gives us twice the horizontal angle α between the crystal surface and the atomic plane. The construction of the crystal holder allows us to insert the crystal having the longest side vertical. We repeat the procedure with the crystal in positions 4c and 4d. The difference in the positions of the tops of the $K\alpha_1$-lines gives us the double vertical angle β between the crystal surface and the atomic plane.

A determination is made of angles α_1, β_1, α_2, and β_2. The crystal plates are removed from both the *A* and the *B* holders and plane parallel glass plates are inserted. By means of a telescope and a scale, the holder *A* is turned angle α_1 in the horizontal direction and β_1 in the vertical direction. Crystal 1 is then inserted. The atomic planes are now parallel to the plane through axes *B* and *H*.

The *B*-crystal is corrected only with regard to the vertical angle β_2.

The spectrometer was tested by determining the glancing angles of the Cu $K\alpha_1$ line in the two first orders. These angles, reduced to 18° C, were

$\varphi_1 = 10^\circ\ 25'\ 59''.2$
$\varphi_2 = 21^\circ\ 13'\ 46''.2$

which give

$d_1 = 4\,244.91$ X.U.
$d_2 = 4\,245.73$ X.U.

in good agreement with earlier measurements.

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